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CONSTRUCTION AND TESTING OF AN ION PHOTOFRAGMENT MOMENTUM SPECT-- TC(U)
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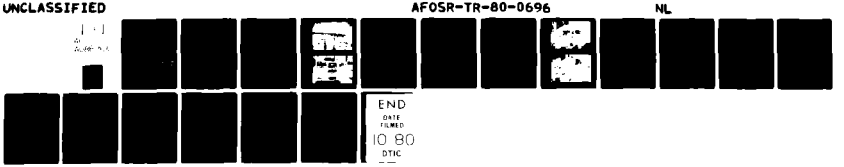

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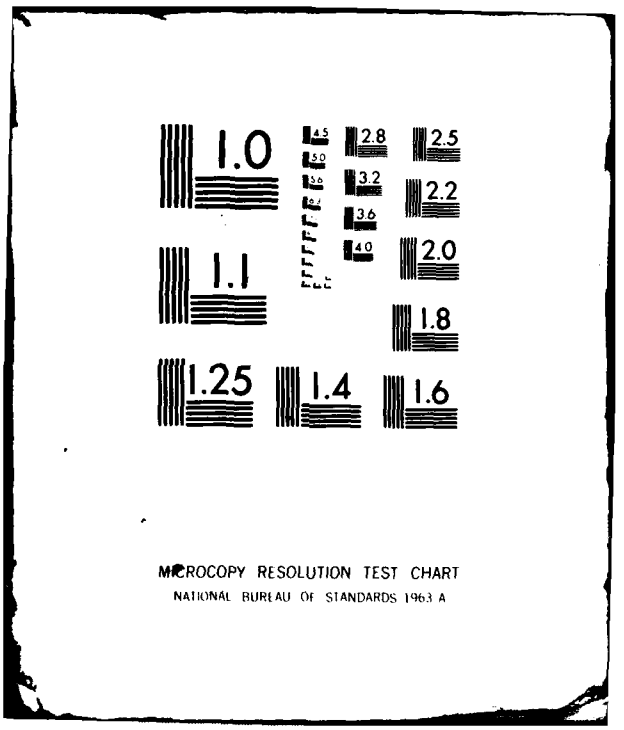
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Ion Photofragment Momentum Spectrometer
N_2O^+
NO^+

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A Nuclide 12-90-G mass spectrometer has been modified for potential use as an ion photofragment momentum spectrometer. It is demonstrated to have the capability of resolving vibrational structure in the momentum distribution of NO^+ produced by photodissociation of N_2O^+ with a detector slit width of 0.25 mm. At this slit width an ion current of 5 picoamps of Ar^+ at the detector is achievable. The operating parameters of the 3-diaphragm single lens used to generate the primary ion beam are presented in detail.

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I. Research Objective

The objective of this project was to modify a Nuclide 12-90-G mass spectrometer so that it would function as a "photofragment momentum spectrometer." The modified instrument was then to be used to determine the distribution of kinetic energies derived from the distribution of momenta of fragment ions formed by photodissociation of D_2^+ and N_2O^+.

II. Status of the Research Effort

In order to convert the existing mass spectrometer into a photofragment momentum spectrometer it was necessary to move the ion source into a new housing and to construct an ion lens which would produce an ion beam focused on (or near) the photodissociation region. The photodissociation region in this case is defined by a 0.635 cm square opening in the side of a stripped-down ion source block, positioned between a bellows-mounted quartz lens and a flange-mounted quartz window (shown near the center of Figure 1).

The ion lens constructed was a "three diaphragm single lens" designed with reference to the data of Hanszen and Lauer.* The lens elements were made from "EV Parts": 304 stainless steel plates, cylinders, and rings; alumina spacers and mounting tubes; and tungsten springs supplied by Kimball Physics, Inc. Figure 3 shows a scale drawing of the ion lens (F1 through F3) positioned between the ion source (IC) and the photodissociation region (PD). Not shown is an adjustable slit (S_1), formerly called the "source slit", which precedes entrance of the ions into the 12", 90° magnetic analyzer. Figures 4a and 4b show the ion lens, plus extraction plate and vertical and horizontal deflection plates, mounted in the new housing.

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* K.-J. Hanszen and R. Lauer, "Electrostatic Lenses" in A. Septier's Focusing of Charged Particles, Academic Press, Vol. I (1967), pp. 272-290.

Figure 1. View of the Nuclide mass spectrometer showing new housing for ion source and ion lens (far right), exit window for light beam passing through photodissociation region (near center), and analyzer magnet (far left).

Figure 2. Main control console of Nuclide 12-90-G mass spectrometer.

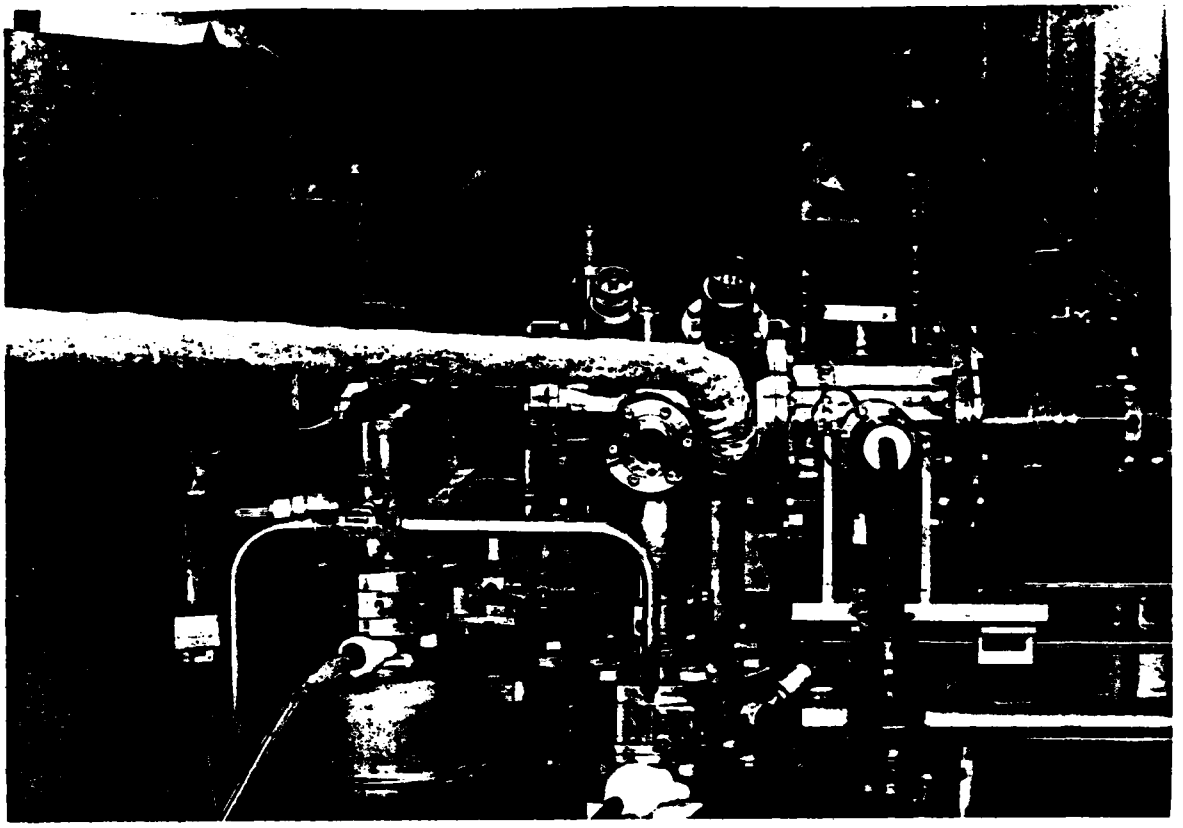


FIGURE 1

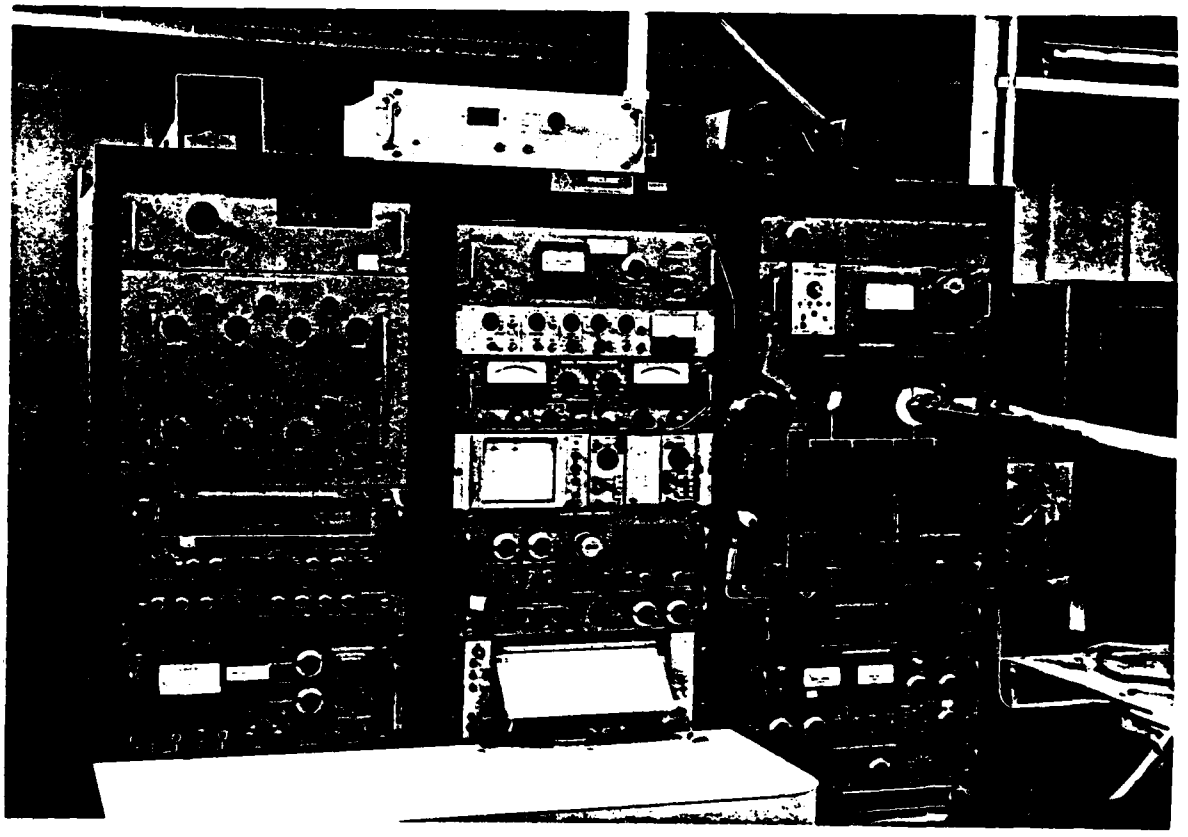


FIGURE 2

Figure 3. Scale Diagram of Ion Optics (Top View)

Rep = Ion Repeller

IC = Ionizing Chamber. Exit slit is .064 cm wide x 0.8 cm high. Dotted circle indicates position of electron beam within ionizing chamber.

EP = Extraction Plate. Aperture is 0.16 cm wide x 1.27 cm high.

F1 = First Focusing Plate. Central aperture is circular, 0.635 cm I.D.

F2 = Second Focusing Plate (with a 1.27 cm long, 1.27 cm O.D., cylinder centered in it)

F3 = Third Focusing Plate (with a 0.635 cm central hole shielded by a 1.27 cm long, 1.27 O.D., cylinder on the "downstream" side)

HORZ = Horizontal steering plates (2 parallel plates)

VERT = Vertical steering plates (2 parallel plates)

PD = Photodissociation region. Exit slit is .046 cm wide x 0.79 cm high.

F4 = Fourth Focusing Plate. The two halves are electrically connected. Used to accelerate ions leaving PD.

F5 = Fifth focusing plate (2 unconnected halves). Used for horizontal steering and additional acceleration f ions.

VERT CENTER = Final vertical steering plate (2 unconnected halves)

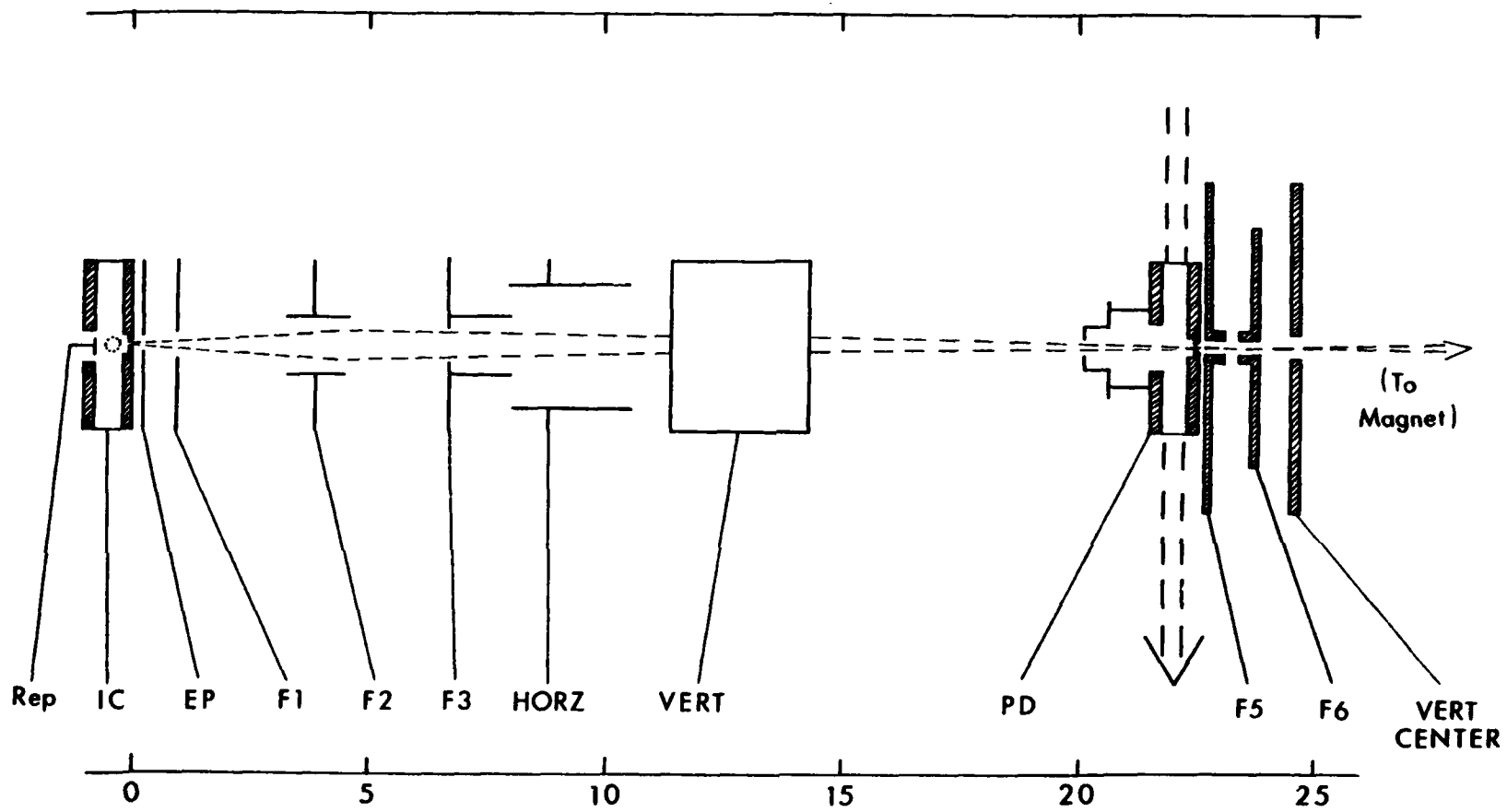

Rep
IC
EP
F1
F2
F3
HORZ
VERT
PD
F5
F6
VERT CENTER
(To Magnet)
0
5
10
15
20
25
DISTANCE IN CENTIMETERS

FIGURE 3

Figure 4a. Ion lens installed in new source housing, showing connections to high voltage feed-through (left side of housing). View is from the source end (new end flange and ion source mounted on it have been removed for this photograph), looking toward the photodissociation region and analyser magnet. The closest plate to the camera was the extraction plate (EP). The stainless steel "cups" above and below the extraction plate hold the source magnets.

Figure 4b. Ion lens installed in new source housing. View is from the side of the magnet. The nearest plates to the camera were the vertical deflection plates.

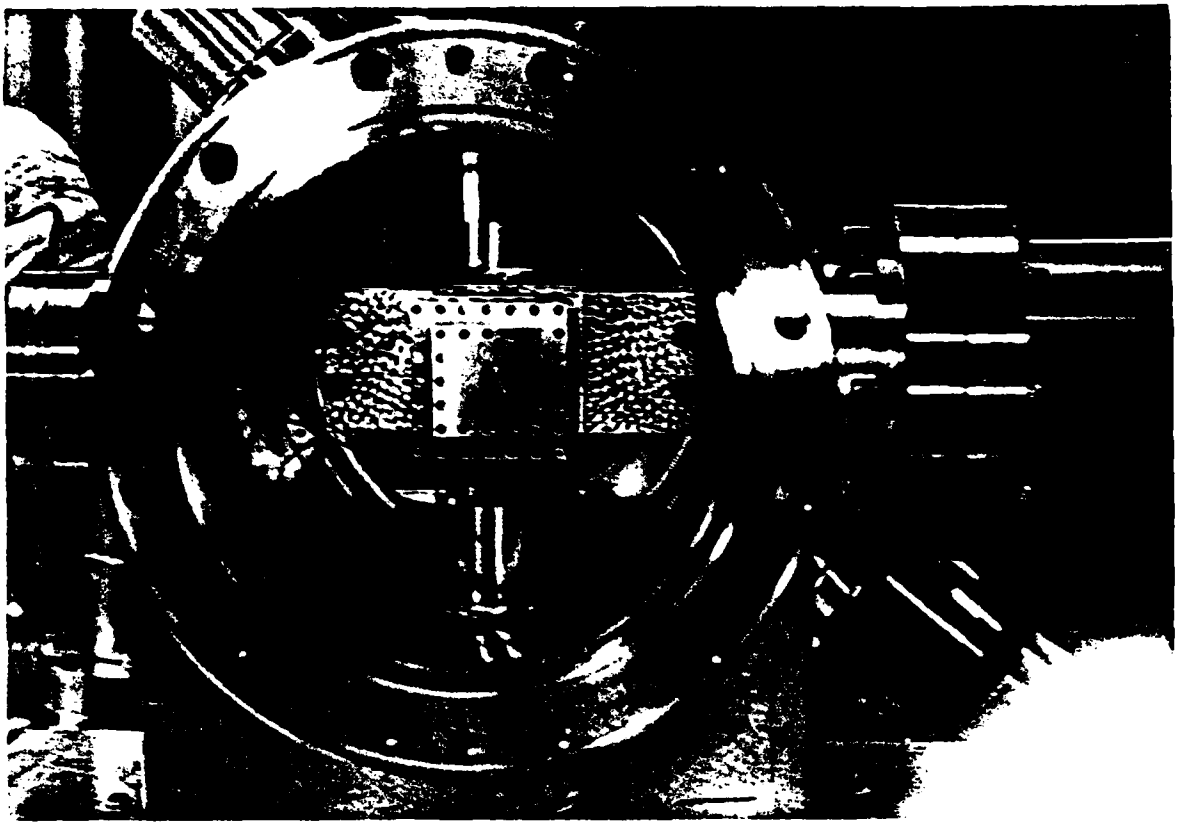


FIGURE 4a

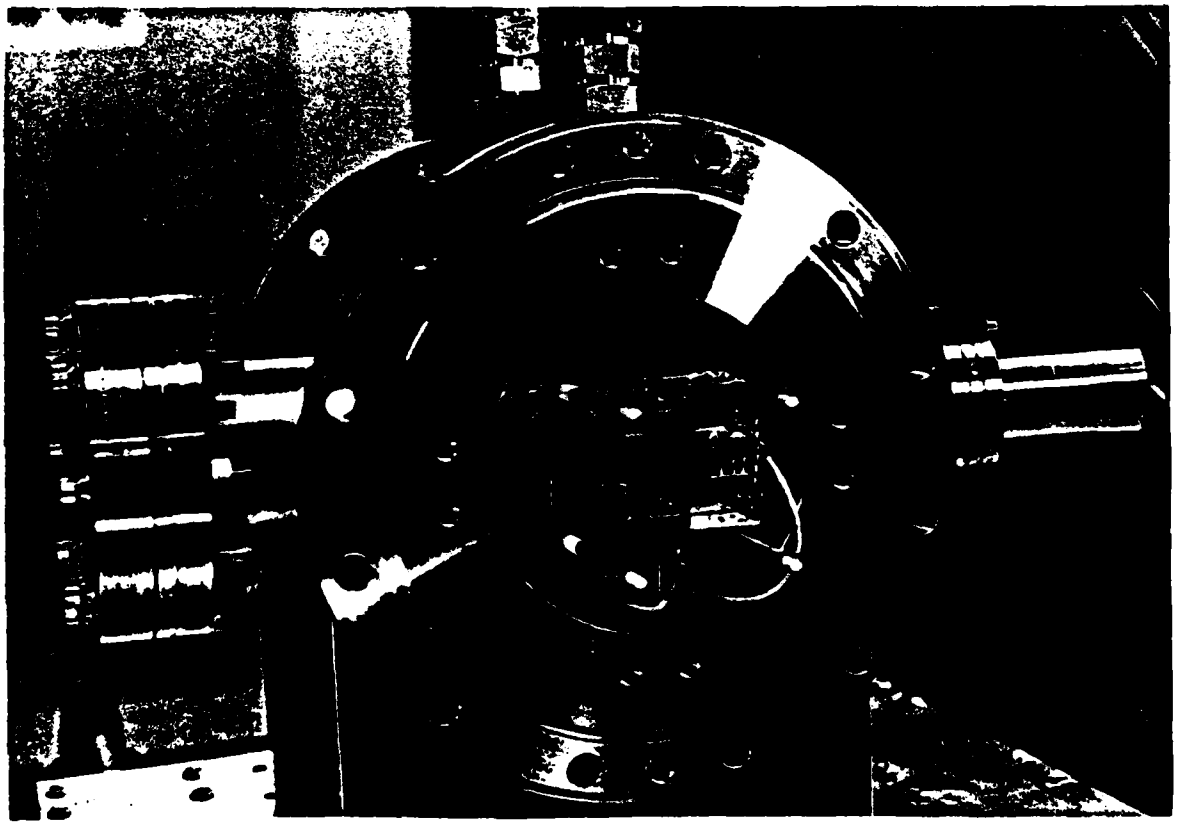


FIGURE 4b

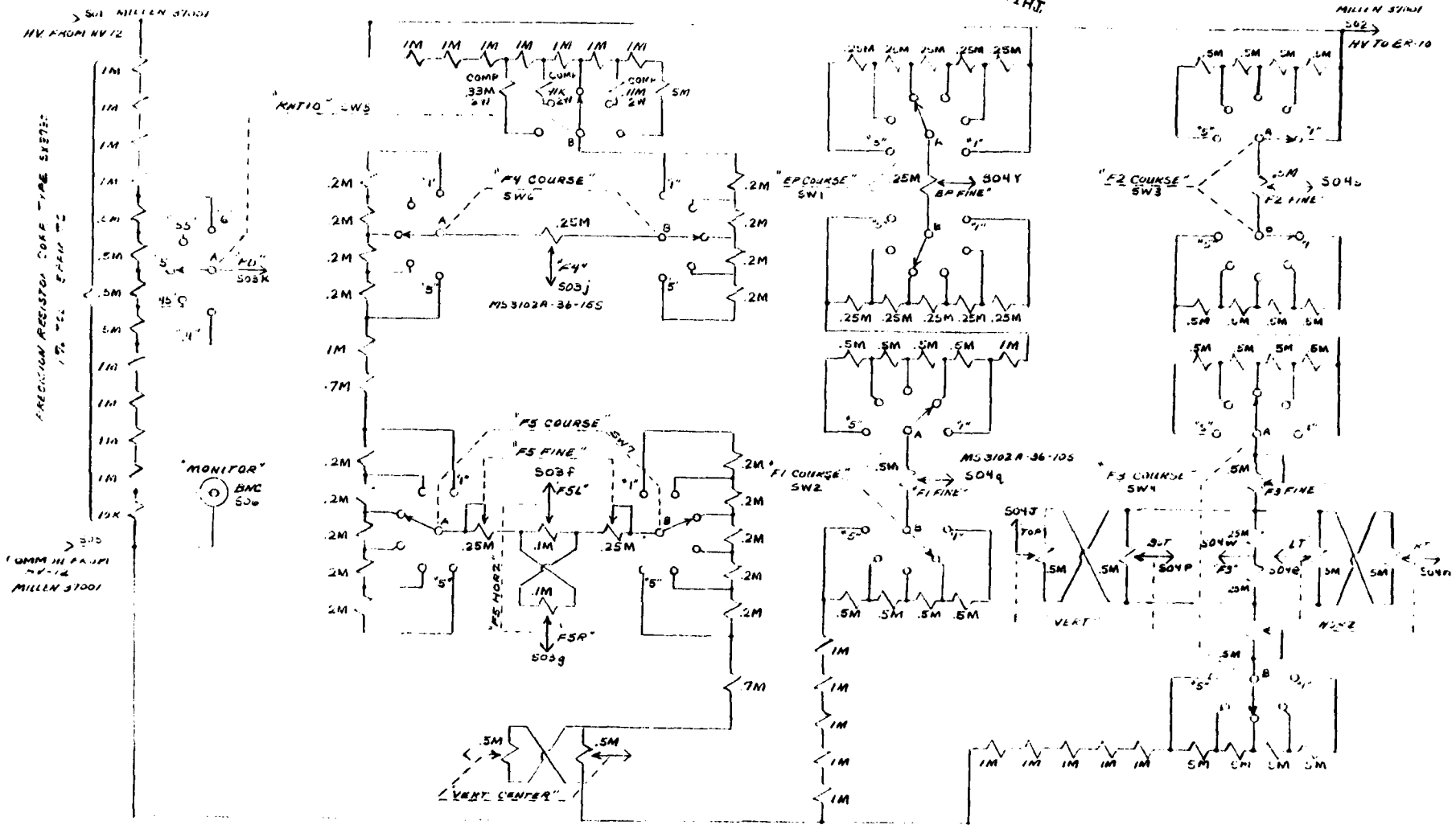
The ion source itself is mounted on a 8¼" end flange, equipped with a 13-pin feedthrough rated at 10 KV, for convenience of changing filaments and cleaning the source. This feedthrough was one cause of delay in carrying out the project; it cracked the second time it was placed under vacuum, and 4-5 months elapsed before the supplier (Nuclide Corp.) completed its repair.

In order to minimize voltage drifts a new high voltage divider was built to supply all the elements shown in Figure 3, rather than just building a unit to supply the ion lens. Figure 5 shows the circuit diagram for the voltage divider. The fixed resistors in this circuit were selected from Dale Electronics, Inc. and Precision Resistor Corp. for their low temperature coefficients. For example the resistors in the string which determines the ratio of the voltage in the photodissociation region to that in the ionizing chamber all have temperature coefficients $\leq 5 \times 10^{-6}/^{o}C$. The use of matched resistors with low temperature coefficients, combined with high voltage and magnet current power supplies regulated to better than 0.01% should assure the necessary ion beam stability to carry out high resolution momentum analyses even if scan times as long as an hour are required.

Figure 2 shows the main control console for the Nuclide 12-90-G mass spectrometer. The new voltage divider is visible in the left rack, second from the top. Directly below that unit is the control circuit for the RPD (retarding potential difference) ion source used in the preliminary tests of the apparatus reported below. The insulated, heatable tube on the far right of Figure 2 carries gas effusing through a gold leak to the ion source, via internal tubulation from the feedthrough on the housing for the photodissociation region (see Figure 1).

Initial tests of the ion beam obtained with the new apparatus were made with an argon sample so that the beam would consist predominantly of one ion, Ar^{+}. The new ion lens showed a strong focusing action. With an ion accelerating potential (IC) of 4 KV the ion current passing through the detector slit (S_d) was maximized by applying the following voltages to the ion optical elements.

Figure 5. High voltage divider circuit for ion lens, photodissociation region, and additional accelerating and steering plates. The DC-1, 1% T-2 resistors made by Dale Electronics have a carbon film deposited on a ceramic core and epoxy coating for moisture protection. They have a precision of 1%, a temperature coefficient of 50 ppm/^{o}C., and are rated at one watt.



ALL RESISTORS TYPE RN-70, ±1%, T-2 EXCEPT AS NOTED
ALL SWITCHES MIDA SWITCH TYPE 86, SHORTING, 30° INDEX

PHOTO DEFLECTION CONTROL DIVIDER

Table I. Optimum Focusing Voltages

IC = + 4000.V, Rep = + 4003.5V

EP	= + 3490.V	PD	= + 2000.V
F1	= + 2740.V	F4	= + 2000.V
F2	= + 3703.V	F5	= + 710.V
F3	= + 2673.V		

From the above voltages on elements F1, F2, and F3 one can calculate the characteristic voltage ratio for a three-diaphragm lens system, as defined by Hanszen and Lauer:

$$(1) \qquad R_{SL} = \frac{U_i - U_o}{U_a - U_o} = 0.23$$

where U_o = rest potential of the ions = + 4000 V

U_a = potential of the outer electrodes

$\sim$ ½(F1 + F3) = 2706 V

U_i = potential of the intermediate electrode

= F2 = + 3703 V

From the lens power curves for symmetrical single lenses presented by Hanszen and Lauer (Fig. 12, p. 275) and the calculated value of R_{SL}, the focal length of our ion lens with the operating voltages shown in Table I is estimated to be $\sim$ 7 cm. This distance is somewhat greater than the distance from the exit slit of the ion source to the center of F2 (3.9 cm) presumably because the extraction plate and the superstructure of the photodissociation region both act as weak converging lenses.

With an argon pressure of $\sim$ 27 Pascals (0.2 Torr) in the sample reservoir behind the $\sim$ 10 micron gold leak and 6.7 x 10^{-4} Pa (5 x 10^{-6} Torr) in the source housing, an ionizing voltage of 50 V and a trap current of 6 x 10^{-7} A, the following ion currents were measured.

Table II. Measured Ion Currents of Ar^+

S_1 (mm)	S_d (mm)	I_{TIM} (A)	I_{FC}(A)
0.30	0.64	4.1×10^{-11}	2.1×10^{-12}
0.20	0.38	2.2×10^{-11}	n.d.
0.10	0.25	1.0×10^{-11}	n.d.
0.04	0.13	3.8×10^{-12}	n.d.

I_{TIM} = Ion current measured by a probe ("total ion monitor") at the entrance to the field of the magnetic analyzer. The probe samples only a fraction of the ion beam.

I_{FC} = Ion current measured with a Faraday cup placed behind the detector slit.

The RPD ion source used to obtain the above ion currents generates a very well-defined electron beam through the collimating and focusing action of 5 small apertures. Trap currents as large as 10^{-5}A can be achieved, which should increase the ion currents in Table II by an order of magnitude. Thus it should be possible to achieve $I_{FC} \sim 5 \times 10^{-12}$A with a detector slit width of 0.25 mm, small enough to resolve the "vibrational structure" in the momentum spectrum of NO^+ produced by photolysis of N_2O^+ (as shown in the research proposal leading to this contract). Taking into account the geometry of the photon-ion interaction region, assuming a light intensity of 1.4×10^{15} photons/sec at 334.1 nm, and a photodissociation cross section for N_2O^+ of 2.6×10^{-18} cm, $\sim 1.8 \times 10^4$ photoproduct ions should pass through the 0.25 mm detector slit per minute. This estimate assumes all NO^+ have the same momentum; a broad distribution of momenta would require a slow scan of the magnetic field lasting many minutes. Since 10^3 ions can be counted with a random error of only 3%, there will clearly be sufficient ions reaching the detector to obtain a resolved photofragment momentum spectrum of NO^+ from N_2O^+.

To check the theoretical resolving power of the mass spectrometer after the modifications described above were completed, a slow, expanded-scale magnetic scan was made from $\frac{m}{z}$ = 38 to 41 for an argon sample. The portion of this scan

around $\frac{m}{z}$ = 40 is shown in Figure 6. The slit widths used are the ones required to resolve the vibrational structure in the NO^+ photofragment momentum spectrum. The mass resolutions calculated in the caption of Figure 6 encompass the theoretical value of 857, which assumes no beam energy spread, no aberrations, etc.

The width of the peak in Figure 6 may also be used to check the potential resolving power of the instrument for photofragment momentum spectroscopy. The basic equation for the system is:

$$(2)\quad B^2 = \left(\frac{2eV_a}{e^2r^2}\right)\left(\frac{m_1^2}{m}\right)\left[\left(\sqrt{\frac{KE}{eV_a}}\,\frac{m_2}{m_1} + 1\right)^2 + \left(\frac{V_b}{V_a}\right)\left(\frac{m}{m_1}\right)\right]$$

where V_a = IC - PD

V_b = PD

KE = the total translational energy divided between the fragments in the photodissociative process

m, m_1, m_2 = masses of ions involved in the reaction:

$$m^+ + h\nu \rightarrow m_1^+ + m_2$$

r = radius of curvature of circular path followed by ions in the magnetic field

B = magnetic flux density in Tesla

For $V_a = V_b$ = 2000 V and KE = 0.90 eV NO^+ ions formed by photodissociation of N_2O^+ will enter the detector slit when B = 0.1509 Tesla. Using $\Delta B_{\frac{1}{2}}/B = 4.2 \times 10^{-4}$ from Figure 6 and $\frac{\partial KE}{\partial B} = 2.0 \times 10^3$ eV/Tesla calculated from equation (2), one obtains $\Delta(KE)_{\frac{1}{2}}$ = 0.13 eV. This predicted peak width is less than half the 0.27 eV spacing between vibrational levels in NO^+.

At the time of writing this report the photon counter (SSR model No. 1110) and associated amplifier/discriminator (SSR No. 1120), which will be adapted to count the photo-product ions, are being used to complete another project. As soon as this equipment can be connected to the electron multiplier of the Nuclide 12-90-G, the photofragment momentum spectrometer will be operational. Since

Figure 6. Magnetic scan of $^{40}Ar^{+}$ Peak. S_1 = 0.010 cm and S_d = 0.025 cm. Peak width corresponds to a mass resolution, $R = \frac{M}{\Delta M}$ = 1033 (at 50% of peak height) or 516 (at 5% of peak height). All operating conditions were as given in Table I. "Total ion current", measured at entrance to magnetic analyzer, was 1×10^{-11} A. Argon pressure $\sim$ 0.2 Torr in reservoir behind $\sim$ 10μ gold leak, 5×10^{-6} Torr in source housing. Ionizing voltage = 50 V. Repeller voltage = + 3.5 V. Trap voltage = + 25 V.

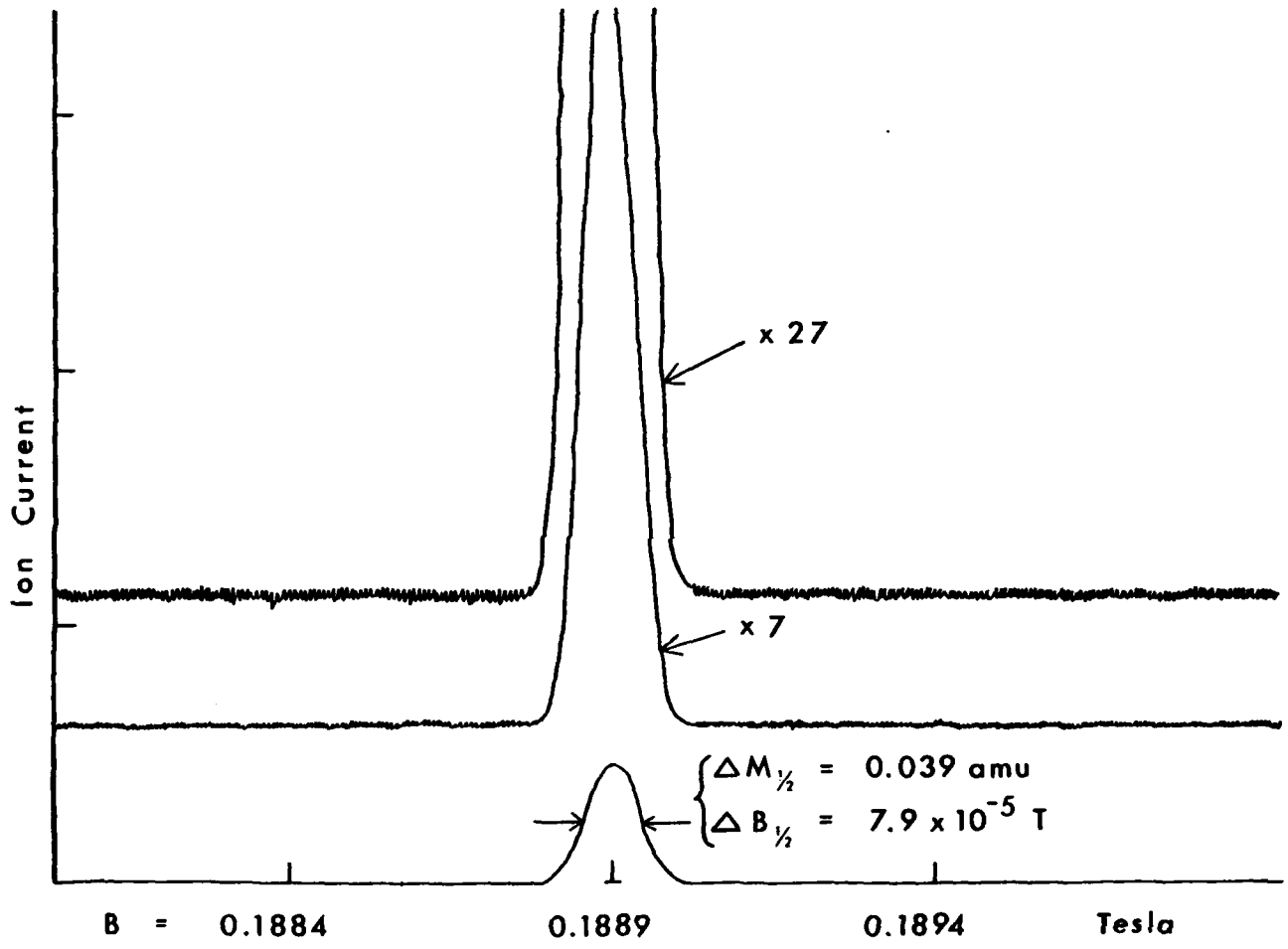

Ion Current
x 27
x 7
ΔM½ = 0.039 amu
ΔB½ = 7.9 x 10⁻⁵ T
B = 0.1884
0.1889
0.1894
Tesla

FIGURE 6

the only light source in place at present is a 1 KW mercury capillary arc, initial experiments will be limited to irradiations at 313.1, 334.1, 365.0, and 435.8 nm (depending on the ion to be studied). Funding for a tunable dye laser, which would: permit selecting λ_{irr}'s corresponding to peaks in the photodissociation spectra for different ions; improve the photo product count rate to background ratio; and enable use of narrower slits for higher resolution in the momentum (or KE) spectrum, is unquestionably needed for fullest utilization of the newly-completed apparatus.

III. Written Publications.

1.) T.F. Thomas, T.L. Rose, and J.F. Paulson, "On the Photochemical Stability of $H_3O^+(H_2O)_n$", J. Chem. Phys., 71, 552 (1979).
2.) K.S. Snow, F. Pallas, and T.F. Thomas, "Vibronic Dependence of Emission Quantum Yields of Glyoxal Vapor", in preparation. Probable journal: Journal of Physical Chemistry.

IV. Assisting Personnel.

1. Mr. Mial Warren, who received a B.A. with majors in chemistry and physics from William Jewell College in May, 1980. Mr. Warren was a research assistant at UMKC from June 18 - August 24, 1979. His principal responsibility was designing and constructing the high voltage divider circuit, although he assisted with other aspects of the project as well. Mr. Warren will begin graduate study in chemical physics at Rice University this fall, supported by an NSF fellowship.
2. Mr. Lloyd Selberg, electronics engineer in the UMKC Chemistry Department. Mr. Selberg finished construction of the high voltage divider, making several modifications and improvements, after the back-ordered precision resistors arrived. Mr. Selberg also made repairs on the electronics of the mass spectrometer, made the necessary high voltage cables and connectors, and generally provided valuable advice and assistance.

V. Acknowledgement

The work done on this project was aided by a UMKC Faculty Research Grant in the amount of $3384, awarded in 1978. A significant portion of this grant was spent on the salary of Mr. Warren and on assorted pieces of high vacuum hardware (flanges, feed-throughs, etc.).